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A Method for Growing Single Crystals of Metallic Indium Antimonide under Pressure

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A method is described for growing single crystals of the metallic β-tin phase of InSb. These crystals were grown from the melt at a pressure of 26 Kbars and recovered, in their metastable state, at liquid-nitrogen temperature. They were cylindrical in shape with lengths ranging from 6–20 mm and diameters ranging from 3–6 mm.

The phase diagram of InSb has been studied by Jayaraman *et al.*[1,2] and Hanneman *et al.*[3] who showed that the transition to metallic InSb occurs near 23 Kbars at room temperature and that the transition pressure depends only slightly on temperature. This solid–solid transition has a large change in volume associated with it ($\approx 20\%$). Jayarman found that the transition was very sluggish at room temperature but became much sharper at higher temperatures. Jamieson[4] subsequently showed that the crystal structure of this phase was very close to, if not identical with, that of white tin. More recently, several people[5–7] have found that the phase of InSb stable at pressures above 30 Kbars has an orthorhombic, rather than β-tin structure.

Darnell and Libby[8,9] have developed a technique for removing InSb, in its metallic β-tin phase, from the high-pressure chamber. This was done by heating and compressing the material well into the region of the P–T diagram where the metallic β-tin phase is thermodynamically stable. The material was then cooled, while under pressure, to liquid-nitrogen temperatures, at which point the pressure was reduced to one atmoshpere and the sample removed. The material so obtained has been identified as having the β-tin structure with lattice parameters essentially identical to those of β-tin.

We will describe in this paper a method for growing single crystals of the metallic β-tin phase of InSb under pressure and recovering them at one atmosphere. The crystals so obtained were cylindrical in shape with lengths ranging from 6–20 mm and diameters ranging from 3–6 mm.

The press used in this work was of the piston-cylinder hydraulic-ram type very similar to the one described by Kennedy and La Mori.[10] A schematic diagram of the pressure chamber with the pressure cell in position is shown in Fig. 1. An aluminum trough was pressed around the outside of the retaining rings and was filled with liquid nitrogen when it was desired to cool the sample.

Figure 2 shows the details about the pressure cell. This figure is self-explanatory with regard to the materials used and their dimensions. The starting material was semiconducting grade n-type polycrystalline indium antimonide obtained from Cominco Products, Inc.

The sample was first molded into a cylindrical shape with one of its ends tapered to a point. The mold material was a high-purity, fine-grained graphite obtained from Poco, Inc. The molding process was done under vacuum using a vertical tube furnace. The sample and mold were then fitted into an insulating cylinder made of talc which in turn was fitted into a cylindrical

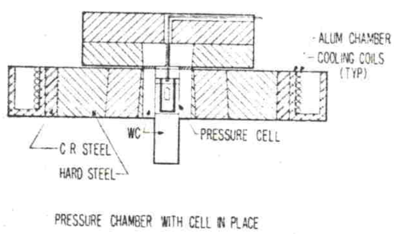

FIG. 1. Press chamber with cell in place.

graphite tube furnace with a tapered wall thickness. This assembly was adjusted so that the pointed end of the sample was near the thick end of the furnace. The furnace was then surrounded with a talc sheath. These components together with a stainless steel cap and pyrophyllite pressure seal constitute the pressure cell assembly. A $\frac{1}{16}$ in. diameter hole was drilled through the steel cap and into the talc so that a chromel–alumel thermocouple, encased in a 2-holed mullite tube, could be placed about 2 mm above the pointed end of the sample. A 5 kVA transformer controlled by a motor-driven powerstat in the primary circuit supplied power to the resistance furnace.

This pressure cell differs from the normally used cell in having a furnace with a tapered rather than uniform wall thickness. The furnace was tapered so as to provide a temperature gradient along its length; the thicker end being at a lower temperature. The sample, upon being cooled from the liquid phase, solidifies first at its pointed

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